



Safe positive temperature coefficient composite cathode for lithium ion battery

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HIGHLIGHTS

- The T_c of PTC material is about 90 °C, much lower than the previously reported.
- They can endow the electrode with a current limiting effect at around 90 °C.
- It will not impose any negative effect on its electrochemical property.
- PTC composite also can be used with LiCoO_2 or $\text{LiCo}_{1/3}\text{Mn}_{1/3}\text{Ni}_{1/3}\text{O}_2$, etc.

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ABSTRACT

Ethylene vinyl acetate (EVA) based positive temperature coefficient (PTC) material with a transition temperature (T_c) of 90 °C is proposed and successfully fabricated in this study. It is further introduced into LiFePO_4 cathode by directly mixing it with LiFePO_4 powder, binder and conductive carbon or sandwiching it between the current collector and LiFePO_4 electrode membrane. Thus obtained LiFePO_4 /PTC composite electrodes both show a self-current-limiting effect at 90 °C. The electrochemical properties of the LiFePO_4 /PTC composite electrodes are determined in terms of galvanostatic charging/discharging, cyclic voltammograms and electrochemical impedance spectroscopy measurements. Comparing with bare LiFePO_4 electrode, both LiFePO_4 /PTC composite electrodes show no degradation in cycling stability, rate capability and electrochemical kinetic property at room temperature. The results indicate that the proposed LiFePO_4 /PTC composite electrode with the suitable T_c of 90 °C can effectively prevent thermal runaway before the occurrence of side reactions and better protect lithium ion battery during the abnormal temperature increasing.

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1. Introduction

Lithium-ion batteries have revolutionized the portable electronics market since it was first proposed in 1990's, but even now, safety concerns associated with the electrode reaction and solid-electrolyte interphase (SEI) instability still hinder the development of lithium ion battery and its wider application. Because of the using of highly flammable organic solvents and the highly reactive electrode material in lithium ion battery, safety risk seems unavoidable in some degree, especially when being used under abusive conditions, lithium-ion batteries may undergo thermal runaway that generates a sharp rise in temperature and then results in serious hazards of fire and explosion. Therefore, it becomes very important to find an effective approach to retard the fire and explosion. A number of strategies have been implemented to improve the safety of lithium-ion batteries, including the addition of safety protection agent and the adoption of the safety

technique, such as fire retardant additive [1], overcharge protector [2], lithium salt [3], and safety vents [4], internal or external PTC devices [5–7], shutdown separators [8], coatings [9], and so on.

Generally, thermal stability of the battery components is a decisive factor in the safety property. Any abuse, including disposing in fire, overcharging, external short circuiting or crushing, can trigger spontaneous heat evolving reactions, which can lead to fire and explosion [10]. And usually, temperature increase is the first abnormal signal that can be detected. Basing on this point, PTC devices such as PTC resistor near the top of the crimped seal header [7], have already been widely used in commercial lithium ion battery. PTC behaviour is characterized by a drastic rise in volume resistivity as temperature approaches the melting point of the matrix resin. The phenomenon is believed to be closely related with localized disaggregation and re-aggregation of conductive fillers as hydrodynamic conditions permit [11]. When PTC device is embedded into lithium ion battery, its sensitivity to temperature increasing can help to introduce a temperature activated switch feature.

However, the distance between the external PTC devices and the SEI, where the thermal runaway most likely starts, can cause some

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hysteresis in PTC's response to temperature. It means that even when the internal cell temperature is above Curie temperature of PTC, the skin temperature of the cell may still not be high enough to activate the external PTC device, and then the effect of PTC device is weakened. The consequence of this hysteresis can be more serious when thermal runaway proceeds quickly and thus more chain reactions may be initiated before the external PTC takes effect. In fact, in the power lithium ion battery, fast charging may cause inhomogeneous polarization and which can further leads to some local temperature increasing within the electrode or in SEI layer and thus the response of the external PTC can be greatly delayed even not able to hinder the electrode reaction before thermal runaways happens.

Reducing the distance between the PTC device and the electrode can be an effective strategy to make PTC device function better, and basing on this, Kise and his coworker proposed a new cathode [5] containing the PTC component, it showed a sharp and nonlinear increase in the electrode resistivity at 130–140 °C due to the expansion of polyethylene at its melting point. Yang reported a new PTC contained cathode [6], in which the PTC layer is sandwiched between the common electrode and the current collector. The result showed that a resistance increasing initiated at around 100 °C, but a complete current-limiting effect required a higher temperature of 130 °C. However, previous work also indicated that at around 100 °C, solvent evaporation, oxidation reaction between the cathode and the electrolyte, or decomposition of SEI already starts [12]. Additionally, it has to be noted that most commercial lithium-ion batteries use Polypropylene/Polyethylene/Polypropylene (PP/PE/PP) separators with a lower melting temperature of 130 °C and the melting of the PE layer at around 130 °C can break the circuit [13]. So, a more effective PTC with lower transition temperature (T_c) is required to better protect the battery before the occurrence of side reaction at 100 °C.

Here we report our recent work on safe electrode containing PTC component, a new EVA-based PTC [14] component was introduced into LiFePO_4 electrode through two approaches, the one is manufacturing the cathode by directly mixing the PTC material with other reagents such as active material, conductive carbon and binder (see Fig. 1a), the other is constructing a sandwich-type electrode by applying a PTC layer between the current collector and the common electrode (see Fig. 1b). Our results show that they both can endow the electrode with a current limiting effect at around 90 °C. It has been further proved that the incorporation of

the PTC component into LiFePO_4 electrode will not impose any negative effect on its electrochemical property, even under a high current rate. Therefore, it is believed that this new PTC/ LiFePO_4 composite electrode is a promising technology for lithium ion batteries with improved safety characteristic.

2. Experimental

The PTC material used in this study is the mixture of polyethylene oxide (PEO), EVA and acetylene black with the mass concentration range of 1–7%, 50–85% and 10–30% respectively, and the typical mixture contained 20 or 24 wt.% acetylene black, depending on how PTC component was introduced into the electrode. Firstly, the above reagents were mixed in a reflux condenser in petroleum ether medium, after 5 h reflux under 105 °C, a well dispersed slurry can be obtained. Thus obtained PTC material is stored into petroleum ether medium for a further use.

The PTC/ LiFePO_4 electrodes were prepared by two methods, one is to mix the above PTC slurry with polytetrafluoroethylene (PTFE) binder, LiFePO_4 powder and acetylene black in the weight ratio of 7.5: 5: 67.5: 20; the other is to construct a sandwich-type electrode by applying a PTC layer between the current collector and the LiFePO_4 electrode membrane, in which the LiFePO_4 electrode contains 75% LiFePO_4 powder, 20% acetylene black and 5% PTFE by weight. A bare LiFePO_4 electrode with the same composition was also made by pressing the above mixture onto the Al foil current collector as a reference. No matter what kind of electrode was used, the size of the electrode is $8 \times 8 \text{ mm}^2$ and the typical loading of LiFePO_4 was 6 mg. The electrode were vacuum dried at 70 °C and then the 2016 cell was fabricated in an argon filled glove box (MECABOX80-1"s", Switzerland) with the above cathodes, lithium anode, 1 M LiPF_6 in 1:1 (v/v) ethylene carbonate (EC)/dimethyl carbonate (DMC) electrolyte, and Celgard 2300 separator.

The charging/discharging tests were conducted on Land battery cycler (Land Co. Ltd., China). Unless specified, the cells were cycled between 2.0 and 4.0 V at C/2 under room temperature ($1C = 150 \text{ mA g}^{-1}$).

Cyclic voltammetry (CV) measurements were carried out on CHI660A Electrochemical Workstation with a scan rate of 0.1 mV s^{-1} . A three electrode cell was used, in which the working electrode has the same composition as being described above, both the counter and the reference electrodes were lithium metal.

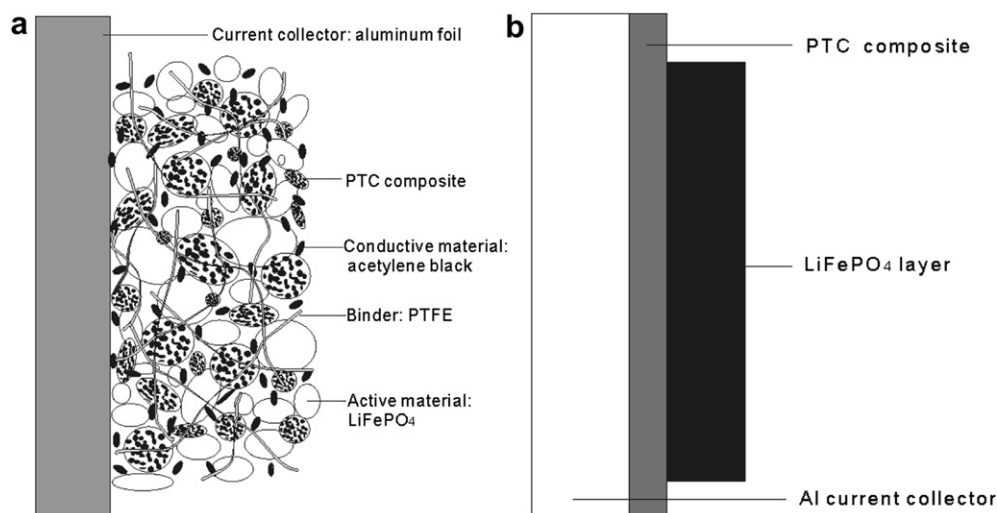


Fig. 1. A schematic illustration of the LiFePO_4 /PTC composite electrode, directly mixture electrode (a) and sandwich-type electrode (b).

AC impedance measurements were performed on an Autolab electrochemical analyser (Autolab PGSTAT30, Eco Chemie) in the frequency range of 100 kHz–100 mHz at an amplitude of 10 mV. The spectrums were all collected on the fresh cells at open circuit voltage (OCV).

The resistivity–temperature characteristics of our PTC material were measured directly by a digital multimeter from ambient temperature (ca. 27 °C) to 110 °C, the method has been widely used in previous works [14,15]. The PTC slurry was poured into a mould, after the evaporation of petroleum ether solvent at 70 °C, a PTC film with the thickness of 0.4 mm was obtained and it was cut into 10×10 mm size and then subjected to the resistance measurement.

3. Results and discussion

3.1. The resistivity–temperature (ρ – T) characteristics of the EVA-based PTC material

Resistivity–temperature behaviour of our EVA-based PTC composites was tested and is shown in Fig. 2. With the temperature increasing, the PTC composite displays four stages of volume resistivity variation in its $\rho(T)$ curve. The first stage is from 27 °C to 62 °C, in which the volume resistivity almost keeps constant. An appreciable change in volume resistivity initiates at 62 °C and continues to 85 °C, in which tens of times of increase in the volume resistivity can be observed. A more dramatic change starts at 85 °C, and from 85 °C to 93 °C, the volume resistivity increases from $4.3 \times 10^4 \Omega \text{ cm}$ to its peak value of $1.7 \times 10^5 \Omega \text{ cm}$ at 92.5 °C. After that, a plateau is observed in the temperature range of 93 °C–110 °C, within it, the volume resistivity decreases slightly, similar to Lee [16], Xiong [17] and Xie's [18] observations. Thus, the PTC intensity of this composite, defined as the ratio of the maximum resistivity to the room temperature resistivity [11], is 463.6. The result suggests that the EVA-based composite may function as an effective switch in lithium ion battery during the temperature variation. Also, it needs to be noted that the T_c temperature of our PTC material is about 90 °C, much lower than the previously reported PTC composite as well as the melting/shutdown temperature of the separator (140 °C), so it is anticipated that it can better protect the battery before the occurrence of the exothermic side reaction.

To ensure a prompt response of the PTC component, PTC material was embedded into LiFePO_4 cathode through two approaches. In the first one, PTC material with the acetylene black content of 20 wt.% were mixed directly with LiFePO_4 powder, PTFE binder and conductive carbon in the fabrication of the LiFePO_4 electrode. Thus obtained cathode is named as DM. In another

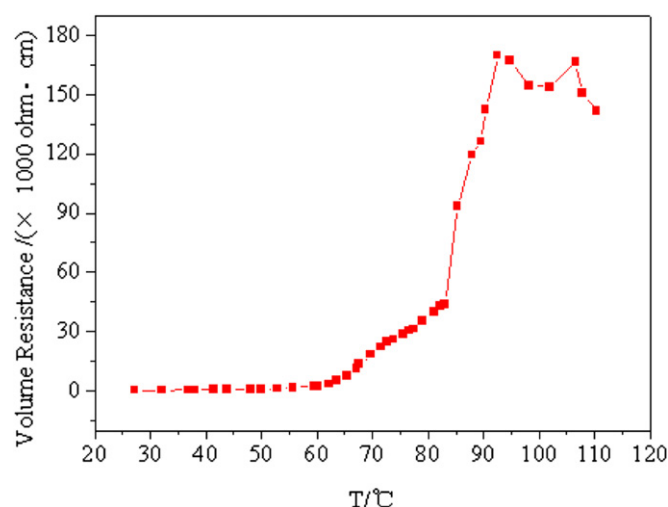


Fig. 2. Dependence of the volume resistance of the EVA-based PTC material on temperature.

approach, the sandwich-type PTC/ LiFePO_4 composite electrode was prepared by firstly applying a thin layer of the PTC material onto an aluminium foil, then pressing an as-prepared LiFePO_4 electrode on it. The typical content of the acetylene black in the PTC layer is 24% and the composite electrode is thus nominated as ST. In order to prove the feasibility of the PTC/ LiFePO_4 composite electrode, we need to make sure that the existence of the PTC component will not affect the electrochemical property of LiFePO_4 electrode, so the cycling behaviour of the above two PTC/ LiFePO_4 composite electrodes is examined.

Fig. 3a compares their cycling stability with that of the bare LiFePO_4 electrode. We can see that, at the room temperature, the capacity delivered at the 100th cycle is almost as same as the bare LiFePO_4 electrode whether for “DM” or “ST” PTC/ LiFePO_4 composite electrode. And in Fig. 3b, the 5th charge/discharge curves are presented together with that of the bare LiFePO_4 electrode, the hysteresis between the charging and discharging plateau of the bare LiFePO_4 , DM electrode and the ST electrode is 0.0946 V, 0.0956 V and 0.0977 V respectively, very close to each other, suggesting their very similar polarization characteristics. The result indicates that the long term cycling behaviour as well as the polarization property under room temperature is substantially determined by LiFePO_4 itself. Therefore, it can be concluded that the introducing of PTC into the LiFePO_4 electrode will not interfere

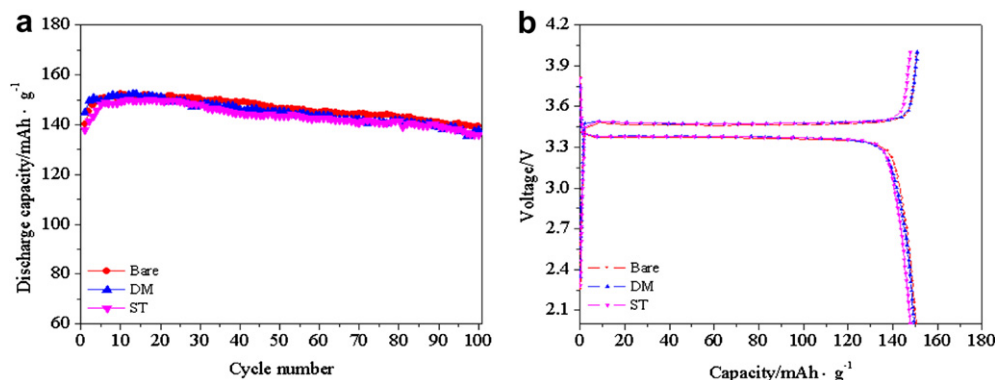


Fig. 3. Comparison of the cycling stability (a) and the 5th charge/discharge profiles (b) of the bare LiFePO_4 , DM electrode and ST electrode at room temperature. (Current: C/2, potential range: 2.0–4.0 V).

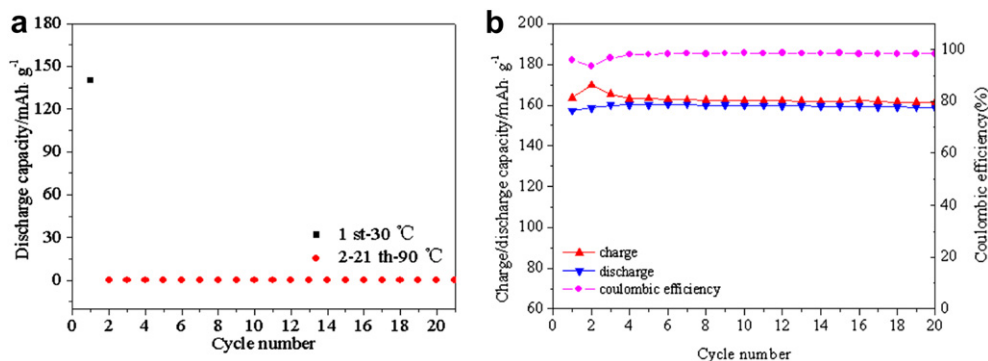


Fig. 4. (a) Capacity performance of the DM electrode at different temperature (1st cycle: room temperature; subsequent cycles: 90 °C); (b) Cycling property of bare LiFePO₄ electrode at 90 °C. (Current: C/2, potential range: 2.0–4.0 V).

with its normal cycling. In the following, the improved safety of the PTC/LiFePO₄ composite electrode will be discussed and investigated.

3.2. Directly mixed electrode

Fig. 4a shows the discharge capacity vs. cycle number plots of the DM cathode at different temperature. The cell were firstly cycled at room temperature, and then it was put into the heating box, whose temperature was set as 90 °C, and its cycling property were tested again. The results show that at 90 °C, the PTC/LiFePO₄ composite electrode is unable to give any capacity. On the contrary, bare LiFePO₄ electrode still cycles well even presenting a higher capacity because of the faster Li⁺ diffusion at high temperature (Fig. 4b). Obviously, the results should be explained by the current-limiting effect brought by the PTC component. In fact, the charging/discharging curve of the PTC/LiFePO₄ composite electrodes further confirms the occurrence of serious ohmic polarization at 90 °C, which is due to the dramatically increased electrical resistance of PTC component and then the whole electrode. The data in Fig. 4 demonstrate that the PTC containing electrodes can serve as a thermal regulator, preventing thermal runaway by raising its resistance, then cutting down the charge transfer within the cathode and lowering its heat generation rate.

In Fig. 5a, the cycling behaviour of the PTC/LiFePO₄ composite electrode at 60 °C is compared with that of bare LiFePO₄ cathode. We can see that the discharging capacity of the PTC/LiFePO₄ composite cathode almost keeps constant as same as bare LiFePO₄ cathode within 20 cycles at 60 °C. However, it is also noticed that at 60 °C, the PTC/LiFePO₄ composite cathode delivers a relatively

lower capacity than the bare one, it should be ascribed to the slightly increased electrical resistance of the PTC component and thus resulted increased polarization. The comparison of the voltage profiles further proves it. In Fig. 5b, at a higher temperature of 60 °C, the voltage difference between the charging and discharging plateau of the PTC/LiFePO₄ composite cathode increased to 0.1562 V, while that of the bare LiFePO₄ electrode is 0.0521 V. In fact, we further optimize the concentration of acetylene black in PTC component, and it has been proved that adjusting the acetylene black concentration can effectively reduce the polarization in high temperature charging/discharging and thus a better capacity performance can be achieved, relative work is still in progress.

Generally speaking, PTC/LiFePO₄ composite cathode can effectively prevent the thermal runaway at 90 °C before the initiation of the side reaction occurring at the SEI, it can well protect the cell without sacrificing the electrochemical property of the cathode at room temperature.

Cyclic voltammogram (CV) profiles of the bare LiFePO₄ and DM electrode, collected at room temperature with a scanning rate of 0.1 mV s⁻¹ are shown in Fig. 6a. A pair of well defined redox peaks can be found for both electrodes, which should be attributed to the Fe³⁺/Fe²⁺ redox couple transformation, accompanied by lithium ion insertion/extraction into and out of the electrode. The CV curves of the two electrodes mostly overlap except a very slight shift in the redox peak as well as an insignificant decrease in the peak current in the DM electrode, but it has to be noted that the potential hysteresis and the peak area of these two electrodes are nearly the same. CV plots obtaining at 60 °C show similar results (Fig. 6b), neither the potential hysteresis nor the peak area changes notably after adding PTC component into LiFePO₄ electrode.

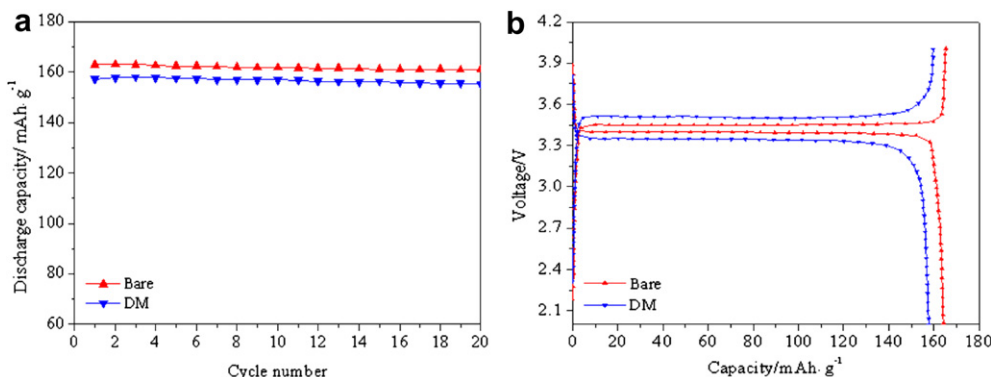


Fig. 5. Comparison of the cycling stability (a) and the 5th charge/discharge profiles (b) of the bare LiFePO₄ and DM electrode at 60 °C.

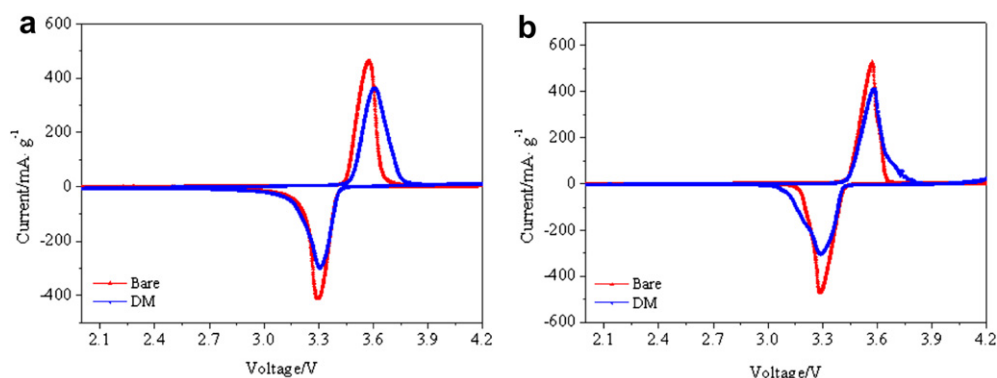


Fig. 6. The cyclic voltammograms of the bare LiFePO₄ and DM electrode at (a) room temperature and (b) 60 °C. (Scan rate: 0.1 mV s⁻¹, potential range: 2.0–4.2 V).

As for practical application, rate performance is very important for LiFePO₄ material. Fig. 7 displays the charge/discharge plots of the bare LiFePO₄ and DM cathode at different current rate, from C/10 to 2 C, under room temperature. For a better comparison, the difference between the average charging potential and the average discharging potential is given in Table 1. From Fig. 7a, we can see that, at all the current rates adopted in the tests, the 1st charge–discharge profiles of the bare LiFePO₄ and DM electrode are almost identical, and the gaps between the charging and discharging plateau are widened in both electrodes with the increasing current rate. The data shown in Table 1 can reveal some difference. From C/10 to C/2, the difference between the average charging potential and discharging potential of the DM electrode is even a little bit smaller than that of the bare LiFePO₄, it can be easily understood if the larger total content of carbon in the DM electrode is taken into account. But when the current rate is further increased to 1 C, the impact of the insulating EVA in PTC component begins to appear, and its increasing influence can be observed with the

increasing current rate, the difference between the average charging potential and discharging potential of the DM electrode increases from 0.0602 V at C/10 to 0.3281 V at 2 C, 13 mV larger than that of the bare LiFePO₄. But even that, the DM electrode still delivers the same capacity as bare LiFePO₄ at 2 C rate. The data further prove that the no significant negative effect on the electrochemical performance can be brought by the introduction of PTC component into LiFePO₄ cathode.

To gain more understanding of the electrochemical properties of the bare LiFePO₄ and DM electrode, AC impedance measurements were conducted. Firstly, their impedance curves were collected at OCV under room temperature, and then the three electrode cells were put into an oven whose temperature was set at 60 °C, and their impedance plots were collected again. The EIS spectra of the bare LiFePO₄ and PTC/LiFePO₄ composite electrode are compared in Fig. 8. It is seen that charge transfer resistance (R_{ct}) of the DM electrode is larger than that of the bare LiFePO₄ electrode at room temperature and 60 °C (Table 2), but the difference is not

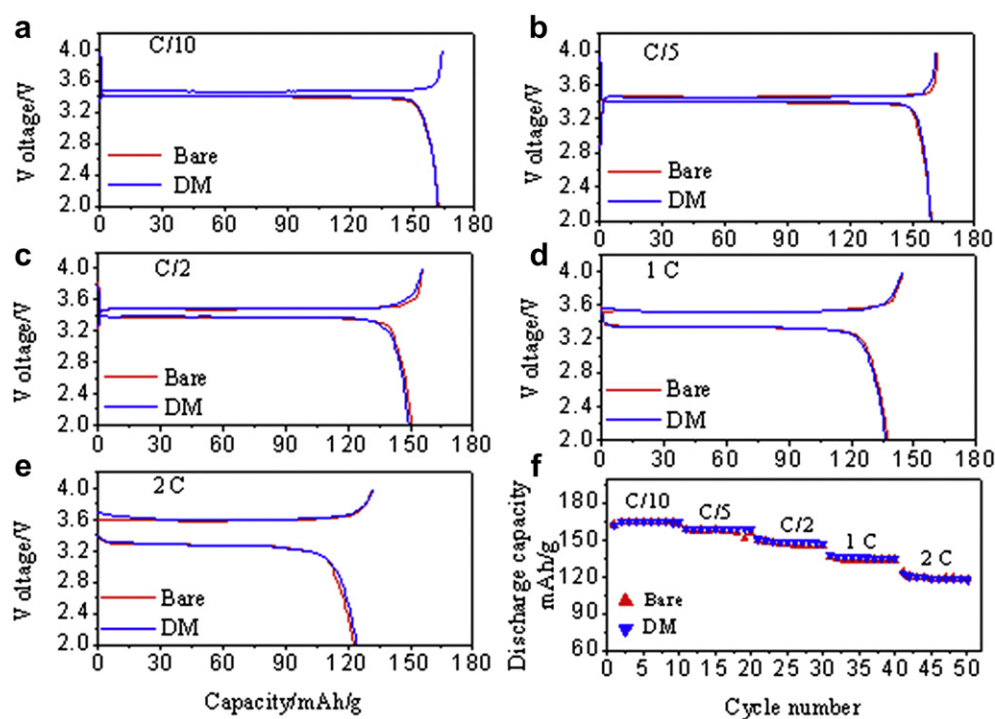
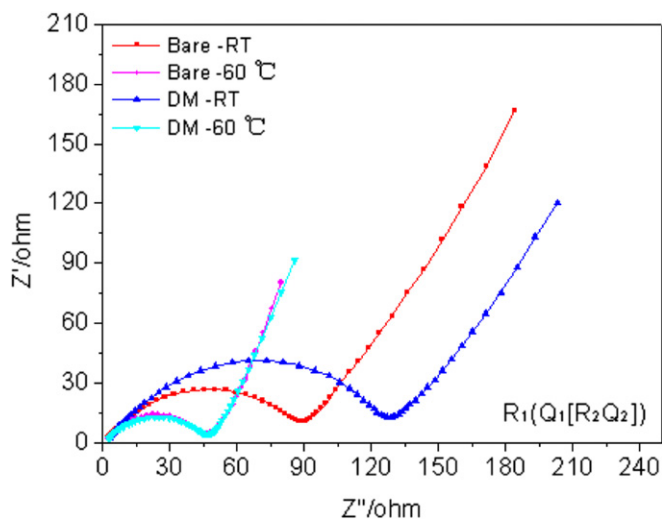


Fig. 7. Charge/discharge profiles of the bare LiFePO₄ and DM electrode at various current rate (a–e) and the comparison of the rate capability of bare LiFePO₄ and DM electrode (f). (Current: C/10, C/5, C/2, 1 C and 2 C, potential range: 2.0–4.0 V).

Table 1

The difference between the charge/discharge voltage plateau of bare LiFePO₄ and DM electrode.

	C/10 1th	C/5 11th	C/2 21th	1 C 31th	2 C 41th
Bare LiFePO ₄	0.0712 V	0.0665 V	0.1022 V	0.1823 V	0.3154 V
DM	0.0602 V	0.0569 V	0.1002 V	0.1830 V	0.3281 V

**Fig. 8.** AC-impedance of the bare LiFePO₄ and DM electrode at different temperature.

significant. The R_{ct} value of the two electrodes both decrease notably at 60 °C, it should be due to the better electrode kinetic property of LiFePO₄ under high temperature [19]. The results from EIS measurement corresponds well with the charge/discharge data shown in the above.

3.3. Sandwich-type electrode

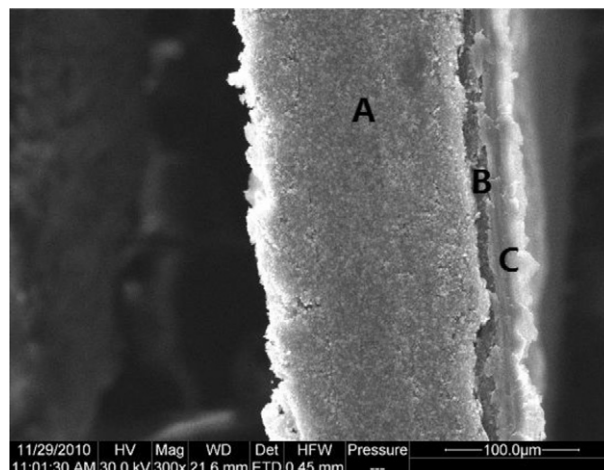
Fig. 9 shows a cross section image of the sandwich-type PTC/LiFePO₄ electrode, in which well resolved PTC layer with the thickness of 10 μm, Al substrate, and the LiFePO₄ electrode layer can be observed.

The discharge capacity vs. cycles at different temperature of the ST electrode is shown in Fig. 10. Obviously, no matter what kind of method is used in introducing PTC component into the LiFePO₄ electrode, the composite electrode will show a self-current-limiting effect when the temperature reaches 90 °C, the T_c temperature of PTC. At room temperature, the ST electrode delivers the capacity of about 140 mAh g⁻¹ in the first two cycles, very close to the capacity released by the bare LiFePO₄ electrode, but when the temperature increases to 90 °C, its capacity drops immediately to almost zero. Therefore, using the sandwich-type PTC/LiFePO₄ composite electrode also can effectively prevent the thermal runaway by stopping the battery reactions triggered by abnormal high temperature.

Table 2

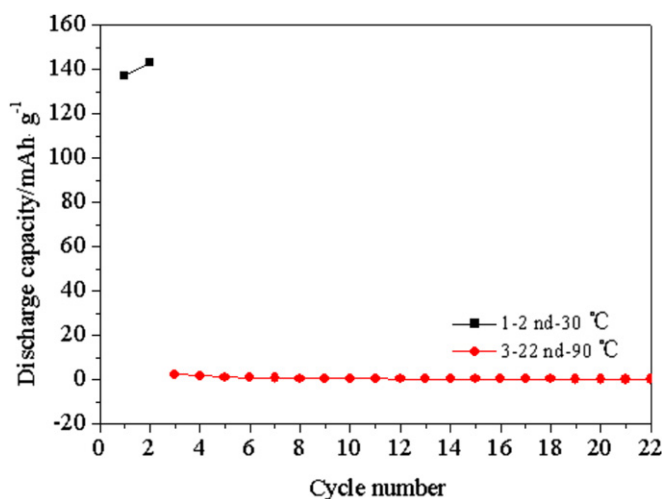
Resistance values obtained by fitting experiment data using the equivalent circuit shown in Fig. 8.

Temperature	Electrode	R_{ct} (ohm)
Room temperature	Bare LiFePO ₄	108.0
	DM	131.7
60 °C	Bare LiFePO ₄	43.8
	DM	47.5

**Fig. 9.** Cross-section image of the sandwich-type (ST) LiFePO₄/PTC composite electrode, (A) LiFePO₄ layer; (B) PTC; (C) Al current collector.

The high temperature (60 °C) cycling stability of the ST electrode and the bare LiFePO₄ electrode is compared in Fig. 11a. At 60 °C, the capacity of the PTC/LiFePO₄ composite electrode and the bare one are both maintained at 160 mAh g⁻¹ or so within 20 cycles. Besides, a further comparison of the 5th charge/discharge curves shown in Fig. 11b, indicates that at 60 °C, the ST electrode shows a little bit larger voltage difference between the charge and discharge plateau (ca. 0.0803 V) than the bare LiFePO₄ electrode (0.0521 V), however, the capacity performance keeps unaffected by the adding of PTC layer between the current collector and the LiFePO₄ electrode.

AC impedance measurements were carried out at room temperature and 60 °C. Fig. 12 compares the impedance spectrum of the sandwich-type PTC/LiFePO₄ electrode and the bare LiFePO₄ one. The plots were all collected under the same condition as being described above. Fitted by the equivalent circuit diagram given in Fig. 12, R_{ct} value can be obtained. We can see that, at room temperature, the ST electrode presents the R_{ct} value very close to that of the bare LiFePO₄ electrode; while at 60 °C, the ST electrode shows a bigger R_{ct} value than bare LiFePO₄ electrode (Table 3). The data shown in Table 3 agree well with the result presented in Fig. 3b and Fig. 11b. Namely, at a higher temperature, such as 60 °C, the PTC

**Fig. 10.** Capacity performance of the ST electrode at different temperature (1st cycle: room temperature; subsequent cycles: 90 °C, current: C/2, potential range: 2.0–4.0 V).

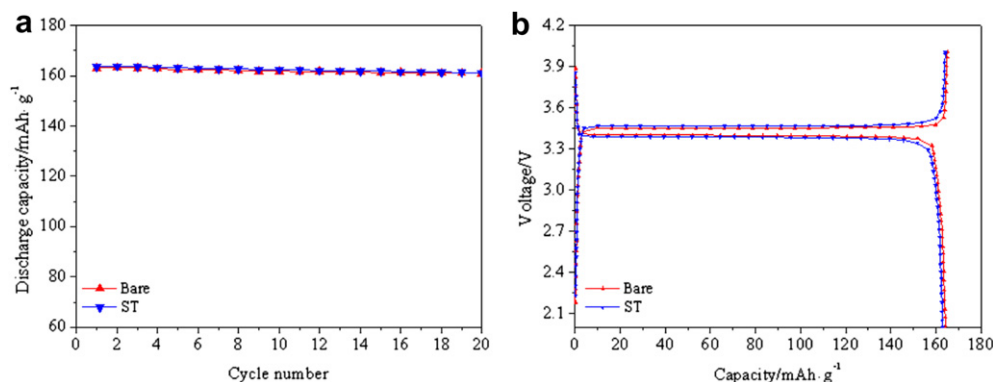


Fig. 11. Comparison of the cycling stability (a) and the 5th charge/discharge profiles (b) of the bare LiFePO₄ and ST electrode at 60 °C. (Current: C/2, potential range: 2.0–4.0 V).

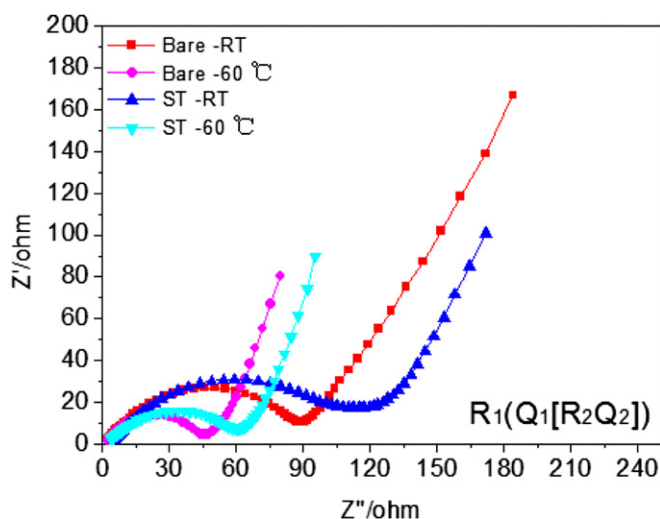


Fig. 12. AC-impedance of the bare LiFePO₄ and ST electrode at different temperature.

Table 3

Resistance values obtained by fitting experiment data using equivalent circuit shown in Fig. 12.

Temperature	Electrode	R_{ct} (ohm)
Room temperature	Bare LiFePO ₄	108.0
	ST	111.3
60 °C	Bare LiFePO ₄	43.8
	ST	64.4

component can cause some insignificant deterioration in the electrochemical kinetics property, but it is also believed that the increased polarization under high temperature resulting from the introduction of PTC component can be greatly reduced by the optimization of the carbon concentration in PTC layer as well as the electrode preparation, such as spreading the PTC layer with more uniform thickness and a better contact between the PTC layer and LiFePO₄ electrode.

4. Conclusion

In this paper, a safe electrode strategy is proposed for lithium ion battery. Different from previously used PTC devices, which are usually an accessory external to the electrode, we try to embed the PTC material into the LiFePO₄ electrode and construct a PTC/LiFePO₄ composite cathode. The new PTC material with lower T_c of

90 °C is introduced into LiFePO₄ electrode through two approaches. One is to directly mix the PTC component with other ingredients of LiFePO₄ powder, conductive carbon and binder; the other is to sandwich a PTC layer between the Al current collector and the LiFePO₄ electrode. Charging and discharging tests indicate that no matter what kind of method is adopted, the obtained PTC/LiFePO₄ composite cathodes both show a self-current-limiting effect when the temperature is increased to above 90 °C. Consequently, when some side reactions occur, thus resulted abnormal temperature increasing can trigger the dramatic resistance increase in the PTC component and then the whole cathode, and in turn the battery reactions will be shut down. Therefore, using PTC/LiFePO₄ composite cathode can effectively prevent a further thermal runaway and supply the protection during the abnormal temperature increasing. CV and impedance measurements reveal that at room temperature, the existence of the PTC component in the LiFePO₄ electrode has no negative influence on its electrochemical property, while at a higher temperature of 60 °C, the slightly increased resistance of the PTC component can cause a slightly increased polarization in the composite electrode, but it is believed that this problem can be solved by the optimization of the concentration of ethylene black in PTC or electrode preparation. Additionally, it can be very reasonably anticipated that basing on the proposed PTC material, other PTC composite cathode, such as PTC/LiCoO₂ or PTC/LiCo_{1/3}Mn_{1/3}Ni_{1/3}O₂, etc, with improved safety can be thus constructed.

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